



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁵ : C11D 1/14, 10/04, 17/00	A1	(11) International Publication Number: WO 91/16409 (43) International Publication Date: 31 October 1991 (31.10.91)
(21) International Application Number: PCT/EP91/00768 (22) International Filing Date: 22 April 1991 (22.04.91) (30) Priority data: 90304440.2 25 April 1990 (25.04.90) AT 90312402.2 14 November 1990 (14.11.90) AT (71) Applicant (for AU CA GB only): UNILEVER PLC [GB/GB]; Unilever House, Blackfriars, London EC4P 4BQ (GB). (71) Applicant (for all designated States except AU CA GB US): UNILEVER N.V. [NL/NL]; P.O. Box 137, NL-3000 DK Rotterdam (NL). (72) Inventors; and (75) Inventors/Applicants (for US only) : DONKER, Cornelis, Bernard [NL/GB]; 3 Jellicoe Close, Caldy, Wirral L48 2LF (GB). MACHIN, David [GB/GB]; 32 Brancote Road, Oxton, Birkenhead L43 6TJ (GB). SCHEPERS, Frederick, Jan [NL/NL]; Albertine Agneslaan 234, NL-3136 NE Vlaardingen (NL).		(74) Agent: TAN, Bian, An; Unilever N.V., Patent Division, P.O. Box 137, NL-3130 AC Vlaardingen (NL). (81) Designated States: AT (European patent), AU, BE (European patent), BR, CA, CH (European patent), DE (European patent), DK (European patent), ES (European patent), FR (European patent), GB (European patent), GR (European patent), IT (European patent), JP, KR, LU (European patent), NL (European patent), SE (European patent), US. Published <i>With international search report.</i> <i>Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i>
(54) Title: LIQUID DETERGENT COMPOSITIONS (57) Abstract An aqueous liquid detergent composition comprising a primary alkyl sulphate material, which is present in a non-solid dispersed phase.		

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AT	Austria	ES	Spain	MG	Madagascar
AU	Australia	FI	Finland	ML	Mali
BB	Barbados	FR	France	MN	Mongolia
BE	Belgium	GA	Gabon	MR	Mauritania
BF	Burkina Faso	GB	United Kingdom	MW	Malawi
BG	Bulgaria	GN	Guinea	NL	Netherlands
BJ	Benin	GR	Greece	NO	Norway
BR	Brazil	HU	Hungary	PL	Poland
CA	Canada	IT	Italy	RO	Romania
CF	Central African Republic	JP	Japan	SD	Sudan
CG	Congo	KP	Democratic People's Republic of Korea	SE	Sweden
CH	Switzerland			SN	Senegal
CI	Côte d'Ivoire	KR	Republic of Korea	SU	Soviet Union
CM	Cameroon	LI	Liechtenstein	TD	Chad
CS	Czechoslovakia	LK	Sri Lanka	TG	Togo
DE	Germany	LU	Luxembourg	US	United States of America
DK	Denmark	MC	Monaco		

LIQUID DETERGENT COMPOSITIONS

The present invention is concerned with aqueous liquid
5 detergent compositions which contain a primary alkyl
sulphate material. More preferably the present invention
relates to aqueous liquid detergent compositions which
contain a primary alkyl sulphate (PAS) material,
optionally in combination with other
10 detergent-active materials and, optionally, sufficiently
dissolved electrolyte to result in a structure of
lamellar droplets dispersed in a continuous aqueous
phase.

15 Primary alkyl sulphates are anionic surfactant
materials, which are especially preferred for use in
detergent compositions for environmental reasons.

A problem in the formulating of liquid detergent com-
20 positions comprising a primary alkyl sulphate as deter-
gent active material is sometimes the occurrence of high
viscosities and/or instability after storage.

It has now been found, that liquid detergent com-
25 positions comprising a primary alkyl sulphate material
and having satisfactory stability and/or viscosity
properties can be obtained by ensuring that the primary
alkyl sulphate is present in a non-solid phase disper-
sed in the system.

30 Accordingly, the present invention relates to an aqueous
liquid detergent composition comprising a primary alkyl
sulphate material, which is present in a non-solid
dispersed phase.

Preferably compositions of the invention are structured liquid detergent compositions. Structuring is especially preferred for providing physical stability to PAS containing compositions. Structured compositions according to the invention may be externally structured (the structuring is provided by materials other than the main active materials) or internally structured (the structuring is provided by main active materials, e.g. detergent active materials in combination with electrolytes). Preferably compositions of the invention are internally structured, more preferably compositions of the invention comprise an internal structure of lamellar droplets of detergent active material, dispersed in the aqueous phase of the composition.

Lamellar droplets are a particular class of surfactant structures which, inter alia, are already known from a variety of references, e.g. H.A.Barnes, 'Detergents', Ch.2. in K.Walters (Ed), 'Rheometry: Industrial Applications', J. Wiley & Sons, Letchworth 1980.

Such lamellar droplet systems are used to endow properties such as consumer-preferred flow behaviour and/or turbid appearance. Many are also capable of suspending particulate solids such as detergency builders or abrasive particles. Examples of such structured liquids without suspended solids are given in US patent 4 244 840, whilst examples where solid particles are suspended are disclosed in specifications EP-A-160 342; EP-A-38 101; EP-A-104 452 and also in US 4 244 840. Others are disclosed in European Patent Specification EP-A-151 884, where the lamellar droplets are called 'spherulites'.

The presence of lamellar droplets in a liquid detergent product may be detected by means known to those skilled in the art, for example optical techniques, various

rheometrical measurements, X-ray or neutron diffraction, and electron microscopy.

5 The droplets consist of an onion-like configuration of concentric bi-layers of surfactant molecules, between which is trapped water or electrolyte solution (aqueous phase).

The primary alkyl sulphate

10

Compositions of the invention comprise as part or all of the detergent active materials a PAS material, preferably a C₈ to C₂₀ PAS, more preferably a C₉ to C₁₅ PAS, most preferably a C₁₀ to C₁₄ PAS. Suitable materials can for example be made by sulphating primary alcohols or are for example available under the trade names Lial 125 ex Enichem, Dobanol 25 ex Shell, Empicol LX ex Albright and Wilson and Texapon LS ex Henkel.

15

20 The level of PAS material is preferably from 0.1 to 40% by weight of the composition, more preferred from 3 to 20%, most preferably from 5 to 15%.

25

The PAS is present in a non-solid dispersed phase. For the purpose of the present invention this means that the PAS material is wholly or predominantly present in a non-solid dispersed phase. Preferably no or only minor amounts (less than 10%, more preferably less than 5%) of the PAS are present in the form of solid crystallites, also preferably no or only minor amounts (less than 10%, more preferably less than 5%) of the PAS are present in solubilised or micellar form.

30

35 The level of PAS in the form of crystallites can be estimated by light microscopy, the level of PAS in solubilised or micellar form can be estimated by

centrifuging the composition at 750G for 16 hours and then determining the level of PAS in the clear layer.

5 Preferably the PAS is predominantly (for more than 80% by weight, more preferably more than 90%, most preferably more than 95%) incorporated in a non-solid dispersed phase. Examples of non-solid dispersed phases are liquid crystal structures, for example lamellar droplet structures. Most preferably the PAS is part of a
10 lamellar droplet structure either as the sole surfactant in the droplets, or more preferably in combination with other surfactant materials.

15 It is believed that it is well within the ability of the skilled person to detect the presence of such a non-solid dispersed phase, for example by light microscopy or electron microscopy.

20 For ensuring that a non-solid dispersed phase comprising the PAS materials is present, several parameters may be varied. Preferably one or more of the following conditions are fulfilled for incorporating the PAS into a non-solid phase:

25 (a) The mole ratio of sodium to other cationic counterions such as potassium and/or ammonium in the composition is from 10 : 1 to 1 : 10, more preferably from 3 : 1 to 1 : 3, most preferably from 2 : 1 to 1 : 2;

30

(b) The PAS material is a branched alkyl sulphate, wherein at least 20%, more preferably more than 30%, most preferably more than 50% of the PAS molecules are branched.

35

- (c) The composition comprises a soap as part of the surfactant system, wherein preferably the soap is predominantly unsaturated. Especially preferably the iodine value of the soap is greater than 70, more preferably greater than 75, most preferably oleate soaps are used.

With respect to condition (a), it is believed that if compositions of the invention contain available counterions for the PAS other than sodium, this will generally lead to less crystallisation of the PAS and therefore generally to a lower viscosity and/or increased stability. Preferably compositions of the invention contain mixtures of sodium and other counterions. Preferably the other counterions are selected from the group of monovalent cations such as potassium, lithium and ammonium, more preferably the other counterions are potassium ions. The mole ratio of sodium to other counterions in the total composition is preferably from 10 : 1 to 1 : 10, more preferably from 3 : 1 to 1 : 3, most preferably from 2 : 1 to 1 : 2.

With respect to (b) preferably the PAS materials are at least partly branched, preferably at least 20% of the PAS molecules are branched, more preferably more than 30%, most preferably more than 50%. Branching is believed to be advantageous because it generally leads to less crystallisation of the PAS materials and therefore to a lower viscosity and/or increased stability.

The use of unsaturated soaps is believed to be advantageous, because this possibly provides a reduced formation of solid complexes of the soap and the PAS material and therefore results in a reduced viscosity and/or an increased stability.

Preferably at least two of the three above mentioned conditions are satisfied, especially conditions (a) and (b), most preferably all three conditions are satisfied.

5 The liquid detergent product

Preferably compositions of the invention are physically stable. In the context of the present invention, physical stability for liquid systems of the invention
10 can be defined in terms of the maximum separation compatible with most manufacturing and retail requirements. That is, the 'stable' compositions will yield no more than 5%, most preferred no more than 2% by volume phase separation as evidenced by appearance of 2
15 or more separate phases when stored at 25°C for 21 days from the time of preparation. Especially preferred are compositions which do not yield any phase separation upon storage for 21 days at 25°C.

20 Preferably, compositions of the invention have a pH of between 6 and 14, more preferred from 6.5 to 13, especially preferred from 7 to 12.

Compositions of the invention preferably have a
25 viscosity after storage for 2 weeks of less than 2,500 mPa.s at 21 s-1, more preferred less than 2,000 mPa.s, most preferred less than 1,500 mPa.s, especially preferred between 100 and 1,000 mPa.s at 21 s-1. Preferably these preferred viscosities are observed
30 after storage for at least two weeks at 25°C, but more preferably also at 15°C and most preferably also at 5°C and 0°C.

Compositions of the invention comprise detergent active
35 materials, preferably at a level of from 1 to 70% by weight of the composition, more preferred a level of 5

to 50% by weight, most preferred from 10 to 40% by weight.

Preferably at least 5% by weight of the detergent active materials are PAS materials, most preferably more than 10%, most preferably more than 20%. Preferably the PAS constitutes less than 60% by weight of the detergent active materials, more preferred less than 50%, most preferred less than 40%.

If for lamellar droplet structured liquid detergent compositions a blend of surfactants is used, the precise proportions of each component which will result in lamellar droplets will depend on the type(s) and amount(s) of the electrolytes, as is the case with conventional structured liquids.

In the widest definition the detergent-active material may, in addition to the PAS material as described above, comprise one or more other surfactants, which may be selected from anionic, cationic, nonionic, zwitterionic and amphoteric species, and (provided mutually compatible) mixtures thereof. For example, they may be chosen from any of the classes, sub-classes and specific materials described in 'Surface Active Agents' Vol.I, by Schwartz & Perry, Interscience 1949 and 'Surface Active Agents' Vol.II by Schwartz, Perry & Berch (Interscience 1958), in the current edition of "McCutcheon's Emulsifiers & Detergents" published by the McCutcheon division of Manufacturing Confectioners Company or in 'Tensid-Taschenbuch', H.Stache, 2nd Edn., Carl Hanser Verlag, München & Wien, 1981.

Suitable nonionic surfactants include, in particular, the reaction products of compounds having a hydrophobic group and a reactive hydrogen atom, for example aliphatic alcohols, acids, amides or alkyl phenols with alkylene oxides, especially ethylene oxide, either alone or with propylene oxide. Specific nonionic detergent compounds are alkyl (C_6-C_{18}) primary or secondary linear or branched alcohols with ethylene oxide, and products made by condensation of ethylene oxide with the reaction products of propylene oxide and ethylenediamine. Other so-called nonionic detergent compounds include long chain tertiary amine oxides, long-chain tertiary phosphine oxides and dialkyl sulphoxides.

Other preferred nonionic surfactant materials are glyceryl ethers such as for example disclosed in GB 1,506,419.

Although compositions of the invention may be free of nonionic surfactants, generally the level of nonionic surfactants is more than 1% by weight of the

composition, preferably from 2.0 to 25.0% by weight of the composition.

Compositions of the present invention may also contain
5 synthetic anionic surfactant ingredients other than PAS. These other synthetic anionic surfactants are preferably used in combination with the above mentioned nonionic materials. Suitable synthetic anionic surfactants are usually water-soluble alkali metal salts of organic
10 sulphonates having alkyl radicals containing from about 8 to about 22 carbon atoms, the term alkyl being used to include the alkyl portion of higher acyl radicals. Examples of suitable synthetic anionic detergent compounds are for example sodium and potassium alkyl
15 (C_9 - C_{20}) benzene sulphonates, particularly sodium linear secondary alkyl (C_{10} - C_{15}) benzene sulphonates; sodium alkyl glyceryl ether sulphates, especially those ethers of the higher alcohols derived from tallow or coconut oil and synthetic alcohols derived from petroleum;
20 sodium coconut oil fatty monoglyceride sulphates and sulphonates; sodium and potassium salts of sulphuric acid esters of higher (C_8 - C_{18}) fatty alcohol-alkylene oxide, particularly ethylene oxide, reaction products; the reaction products of fatty acids such as coconut
25 fatty acids esterified with isethionic acid and neutralized with sodium hydroxide; sodium and potassium salts of fatty acid amides of methyl taurine; alkane monosulphonates such as those derived by reacting alpha-olefins (C_8 - C_{20}) with sodium bisulphite and those
30 derived from reacting paraffins with SO_2 and Cl_2 and then hydrolyzing with a base to produce a random sulphonate; and olefin sulphonates, which term is used to describe the material made by reacting olefins, particularly C_{10} - C_{20} alpha-olefins, with SO_3 and then
35 neutralizing and hydrolyzing the reaction product. The preferred anionic detergent compounds are sodium (C_{11} - C_{15}) alkyl benzene sulphonates.

Generally the level of non-soap, non-PAS anionic surfactant materials is from 0-35% by weight of the composition, for example from 0.5 to 25%.

- 5 It is also possible, and sometimes preferred, to include an alkali metal soap of a mono- or di-carboxylic acid, especially a soap of an acid having from 12 to 18 carbon atoms, for example oleic acid, ricinoleic acid, and fatty acids derived from castor oil, rapeseed oil,
10 groundnut oil, coconut oil, palmkernel oil, alk(en)yl succinates e.g. dodecyl succinate or mixtures thereof. The sodium or potassium soaps of these acids are preferably used. Preferably the level of soap in compositions of the invention is from 0-40% by weight of
15 the composition, more preferred from 5-25%.

- As stated above, preferably the soap material is predominantly unsaturated and has a iodine value of at least 70 more preferably more than 75, most preferably
20 the soap consists for at least 70% by weight of unsaturated soap materials such as oleate.

- Preferably the weight ratio of anionic surfactants (including the PAS material and the soap material) to
25 the above mentioned nonionic surfactant materials is from 10 : 1 to 1 : 10, more preferred from 5 : 1 to 1 : 2, most preferred from 4 : 1 to 1 : 2.

- Also possible is the use of salting out resistant active
30 materials such as for example described in EP 328 177, especially the use of alkyl poly glycoside surfactants such as for example disclosed in EP 70 074. Also alkyl mono glucosides may be used. Preferred levels of these materials are from 0-20% by weight, more preferably from
35 1 to 15%.

The compositions optionally also contain electrolyte, preferably in an amount sufficient to bring about lamellar droplet structuring of the detergent-active material. Preferably the compositions contain from 1% to 5 60%, especially from 2 to 45% of a salting-out electrolyte. Salting-out electrolyte has the meaning ascribed to in specification EP-A-79 646. Optionally, some salting-in electrolyte (as defined in the latter specification) may also be included. In selecting the 10 appropriate level of salting-out electrolyte it is sometimes preferred to use relatively low amounts of salting-out electrolytes, say from 2-10% by weight. These levels are generally sufficiently high to provide structuring of the composition, and do not provide 15 viscosity problems.

In any event, it is preferred that compositions according to the present invention include detergency builder material, some or all of which may be 20 electrolyte. In this context it should be noted that some detergent active materials such as for example soaps, also have builder properties.

Examples of phosphorous-containing inorganic deter- 25 gency builders include the water-soluble salts, especially alkali metalpyrophosphates, orthophosphates, polyphosphates and phosphonates. Specific examples of inorganic phosphate builders include sodium and potassium tripolyphosphates, phosphates and 30 hexametaphosphates. Phosphonate sequestrant builders may also be used. Sometimes it is however preferred to minimise the amount of phosphate builders.

Examples of non-phosphorus-containing inorganic 35 detergency builders, when present, include water-soluble alkali metal carbonates, bicarbonates, silicates and crystalline and amorphous aluminosilicates. Specific

examples include sodium carbonate (with or without calcite seeds), potassium carbonate, sodium and potassium bicarbonates, silicates and zeolites.

- 5 Examples of organic detergency builders, when present, include the alkaline metal, ammonium and substituted ammonium polyacetates, carboxylates, polycarboxylates, polyacetyl carboxylates and polyhydroxysulphonates. Specific examples include sodium, potassium, lithium,
10 ammonium and substituted ammonium salts of ethylenediaminetetraacetic acid, nitrilitriacetic acid, oxydisuccinic acid, melitic acid, benzene polycarboxylic acids, CMOS, tartrate mono succinate, tartrate di succinate and citric acid. Citric acids or
15 salts thereof are preferred builder materials for use in compositions of the invention.

- Preferably the level of non-soap builder material is from 5-40% by weight of the composition, more preferred
20 from 5 to 25% by weight of the composition.

- In the context of organic builders, it is also desirable to incorporate polymers which are only partly dissolved, in the aqueous continuous phase as described in
25 EP 301.882. Typical levels are from 0.5 to 4.5% by weight.

- It is further possible to include in the compositions of the present invention, alternatively, or in addition to
30 the partly dissolved polymer, yet another polymer which is substantially totally soluble in the aqueous phase. Use of such polymers is generally described in our EP 301,883. Typical levels are from 0.5 to 4.5% by weight.

- 35 Apart from the ingredients already mentioned, a number of optional ingredients may also be present, for example lather boosters such as alkanolamides,

particularly the monoethanolamides derived from palm
kernel fatty acids and coconut fatty acids, lather
depressants, oxygen-releasing bleaching agents such as
sodium perborate and sodium percarbonate, peracid bleach
5 precursors, chlorine-releasing bleaching agents such as
trichloroisocyanuric acid, inorganic salts such as
sodium sulphate, and, usually present in very minor
amounts, fluorescent agents, perfumes, enzymes such as
proteases, amylases and lipases (including Lipolase
10 (Trade Mark) ex Novo), anti-redeposition agents,
germicides and colourants.

A preferred further ingredient -which is especially
preferred for incorporation in internally structured
15 compositions according to the invention- is a
deflocculating polymer e.g one having a hydrophilic
backbone and at least one hydrophobic side chain. Such
polymers are described in our copending British patent
applications 8924479.2, 8924478.4 and 8924477.6 and in
20 our European patent application EP 346 995. Typical
levels of these ingredients are from 0.5 to 4.5% by
weight.

Compositions of the invention may be prepared by any
25 conventional method for the preparation of liquid
detergent compositions. A preferred method involves the
dispersing of the electrolyte ingredient -if any-
together with the minor ingredients except for the
temperature sensitive ingredients -if any- in water of
30 elevated temperature, followed by the addition of the
builder material- if any-, the detergent active
materials (preferably as a non-aqueous premix containing
the PAS in acid-form) under stirring and thereafter
cooling the mixture and adding any temperature sensitive
35 minor ingredients such as enzymes perfumes etc. The
deflocculating polymer -if any- may advantageously be

added after the electrolyte ingredients, the builder ingredients or just before cooling.

5 A particularly preferred method of making a aqueous liquid detergent composition which contains a PAS material is to use a premix of PAS with nonionic surfactants, said premix containing little or no water. Preferred premixes contain 10 - 50% by weight more preferably 15 - 40% most preferably 20 - 35% of PAS, and
10 50 - 90% more preferably 60 - 80% of nonionic surfactant. The water level is preferably less than 20% by weight, more preferably less than 10% most preferably the premix is substantially free of water. The PAS in
15 the premix may be present in acid form, but generally the PAS will be present in salt form for example in its sodium salt form.

The premixes can easily be handled and do not suffer from high viscosities. They can for example be supplied
20 as a feedback raw material from as sulphonation or sulphation plant.

In use the detergent compositions of the invention will be diluted with wash water to form a wash liquor for
25 instance for use in a washing machine. The concentration of liquid detergent composition in the wash liquor is preferably from 0.05 to 10%, more preferred from 0.1 to 3% by weight.

30 The invention will now be illustrated by way of the following Examples.

EXAMPLE I

The following compositions were made by adding the ingredients in the listed order to the water at a temperature of 40 °C.

	COMPOSITION % wt	A	B	C	D	E	F
	Sodium citrate.2H ₂ O	10	10	10	10	10	5
	Glycerol	5	5	5	5	5	5
10	Borax	3.5	3.5	3.5	3.5	3.5	3.5
	Fluorescer	0.1	0.1	0.1	0.1	0.1	0.1
	NaOH	2.8	2.8	-	2.8	2.8	-
	KOH	--	--	3.9	--	--	3.9
	polymer ¹⁾	1.0	1.0	1.0	1.0	1.0	1.0
15	Synperonic A7	20	20	20	20	20	20
	Oleic acid	6	6	6	6	10	10
	Coconut fatty acid	4	4	4	4	--	--
	Dobanol 91 PAS ²⁾	10	--	--	--	--	--
	Lial 145 PAS ³⁾	--	10	10	--	10	10
20	Empicol PAS ⁴⁾	--	--	--	10	--	--
	Perfume	0.4	0.4	0.4	0.4	0.4	0.4
	Antifoam (DB100)	0.1	0.1	0.1	0.1	0.1	0.1
	water	<-----balance----->					

25 notes:

1) polymer A-11 as described in EP 346 995 (100%)

2) A C₉-C₁₁ linear PAS ex Shell (as 100%, sodium salt)

3) A C₁₄-C₁₅ PAS being 60% branched ex Enichem (as 100%, sodium salt)

4) A coconut PAS ex A&W (as 100% ammonium salt).

The following product forms were obtained after storage for 2 weeks at the temperature indicated (L=liquid indicates a viscosity at 21 s⁻¹ of less than 2,500 mPa.s, P = Paste indicates a viscosity of more than 2,500 mPa.s at 21 s⁻¹):

COMPOSITION	A	B	C	D	E	F
at 25 °C	L	L	L	L	L	L
at 15 °C	L/P	P	L	L	L	L
at 5 °C	P	P	P	P	P	L
at 0 °C	P	P	P	P	P	L

This example illustrates that PAS containing

compositions of satisfactory viscosity after storage at

25 °C can be obtained by using a Dobanol 91 or Lial 145

PAS (compositions A and B). Further viscosity benefits

can be obtained by using mixed counterions (Compositions

C and D) or by using unsaturated soap materials

(Composition E), especially advantageous results can be

obtained by using mixed counterions in combination with

unsaturated soaps (Composition F).

EXAMPLE II

The following compositions were made as in example I

COMPOSITION % wt	A	B	C	D	E
Glycerol	5	5	5	5	5
Borax	3.5	3.5	3.5	3.5	3.5
Fluorescer	0.1	0.1	0.1	0.1	0.1
Nacitrate.2aq	--	--	--	10	--
Citric acid.1aq	7.1	7.1	7.1	--	7.1
NaOH	4.7	2.4	--	--	2.4
KOH	3.3	6.6	10	--	6.6
polymer ¹⁾	1.0	1.0	1.0	1.0	1.0
Synperonic A7	20	20	20	16	20
Oleic acid	10	10	10	--	10
Lial 145 ³⁾	10	10	10	24	10
water	<-----balance----->				

¹⁾ polymer A-11 as described in EP 346 995 (100%)

³⁾ A C₁₄-C₁₅ PAS being 60% branched ex Enichem (as 100%, sodium salt)

The following product forms were obtained after storage for 2 weeks at the temperature indicated (L=liquid indicates a viscosity at 21 s⁻¹ of less than 2,500 mPa.s, P = Paste indicates a viscosity of more than 2,500 mPa.s at 21 s⁻¹):

COMPOSITION	A	B	C	D	E
at 20°C	L	L	L	L	L
at 4°C	L	L	L	P	L
at 0°C	P	L	L	P	L

This example illustrates that the viscosity after storage is less, when more potassium counterions are present (Compositions A-C). From compositions D-E it follows that higher levels of PAS may cause viscosity problems after storage.

EXAMPLE III

The compositions were made in the following way:

A premix of PAS with nonionic surfactants was made. This premix had a water content of about 6%. Oleic acid was mixed with the premix.

Borax and citric acid and KOH were added to water under stirring followed by the addition of the Zeolyte, polymer, antifoam, the above active mix and the remaining ingredients. Oleic acid could equally well be added separately after addition of the blends.

	COMPOSITION % wt	A	B	C
	Glycerol	4.1	4.1	4.1
	Borax (10 aq)	2.8	2.8	2.8
5	Fluorescer	0.1	0.1	0.1
	Nacitrate (anh.)	6.7	6.7	6.7
	NaOH	1.2	1.2	1.2
	KOH	4.2	4.2	4.2
	Zeolite (4A type)	15.0	15.0	15.0
10	Polymer ¹⁾	0.8	0.8	0.8
	Antifoam	0.2	0.2	0.2
	Nonionic ⁵⁾	17.2	15.4	11.6
	Lial 123 ⁶⁾	4.6	6.4	10.1
	Oleic acid	6.3	6.3	6.3
15	Perfume	0.3	0.3	0.3
	Water	<-----balance----->		

1) polymer A-11 as described in EP 346 995 (100%).

5) Syperonic A7 and Syperonic A3 ex ICI in a 50 : 50
20 weight ratio mixture.

6) A C12-C13 PAS being 60% branched ex Enichem.

Of the above formulations the initial viscosity and the
viscosity after 2 weeks was measured in mPa.s at 21 s⁻¹
25 at ambient temperature was measured, the softening
temperature was measured and the Mole ratio of potassium
to sodium was calculated. The following results were
obtained:

	COMPOSITION	A	B	C
30	NI / PAS ratio	3.7/1	2.4/1	1.15/1
	Mole ratio (K / Na)	1.23/1	1.12/1	0.95/1
	Softening temperature (°C)	< 0	< 0	< 0
	Initial viscosity (mPa.s, 21 s ⁻¹)	815	770	1150
35	Viscosity after 2 weeks of storage at 20°C	790	630	695

This example indicates that satisfactory viscosities of
PAS-containing compositions can be obtained by using
40 mixtures of non-ionics with PAS in different ratios,
together with Zeolite.

EXAMPLE IV

The compositions were made in the following way:

The ingredients were mixed in the listed order, with PAS
5 added as the last active, followed by 20 minutes mixing.

	COMPOSITION % wt	A	B
	Glycerol	5	5
	Borax	3.5	3.5
10	Citric acid	6.4	6.4
	Fluorescer	0.1	0.1
	NaOH	1.1	1.1
	KOH	4.9	4.9
	Polymer	1.0	1.0
15	Syperonic A7	20	20
	Oleic acid	10	10
	PAS ⁷⁾	4	6
	Perfume	0.4	0.4
20	Water	<-----balance----->	

7) Empicol LXV, a sodium coconut PAS ex A&W.

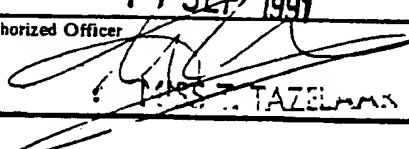
	COMPOSITION	A	B
	NI/PAS ratio	5	3.7
25	Mole ratio (K/Na)	1.26	1.15
	Softening temperature (°C)	< 0	5
	Initial viscosity (mPa.s, 21 s ⁻¹)	240	270
30	Viscosity after 2 weeks of storage at 20°C	195	195

This example shows that also with using a natural
derived material, satisfactory compositions can be
obtained.

CLAIMS

1. An aqueous liquid detergent composition comprising a primary alkyl sulphate material, which is present in a non-solid dispersed phase.
2. An aqueous liquid detergent composition according to claim 1, being structured, preferably internally structured.
3. A composition according to claim 2 wherein the alkyl sulphate material is present in a lamellar droplet phase.
4. A composition according to one or more of the preceding claims, wherein the mole ratio of sodium to other cationic counterions in the composition is from 10:1 to 1:10.
5. A composition according to claim 4, wherein the other cationic counterions are selected from potassium and ammonium.
6. A composition according to claim 1, wherein the PAS material is a branched alkyl sulphate, wherein preferably more than 20% of the PAS molecules are branched.
7. A composition according to one or more of the preceding claims, wherein the composition also comprises a soap and wherein preferably the soap is predominantly unsaturated.

8. An aqueous detergent composition according to claim 1 comprising:
 - a) from 1-70% by weight of detergent active material (including the primary alkyl sulphate);
 - b) from 1-60% of a salting out electrolyte;
 - c) from 0.1 to 40% of a primary alkyl sulphate;
 - d) from 0 to 4.5% of a deflocculating polymer;
9. An aqueous detergent composition according to one or more of the preceding claims, having a viscosity of less than 2.5 Pa.s at 21 s^{-1} after storage for two weeks at 25°C.
10. Use of a composition according to claim 1 for the washing of fabrics.
11. Method of preparing an aqueous detergent composition comprising a primary alkyl sulphate material, said method comprising the mixing of the primary alkyl sulphate material with one or more of the ingredients of the compositions followed by the addition of the remaining ingredients, wherein the primary alkyl sulphate material is added to the compositions as a premix comprising 10 - 50% of primary alkyl sulphate material, 50 - 90% of nonionic surfactant and 0 - 20% of water.

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) ⁶		
According to International Patent Classification (IPC) or to both National Classification and IPC C 11 D 1/14 C 11 D 10/04 C 11 D 17/00		
II. FIELDS SEARCHED		
Minimum Documentation Searched ⁷		
Classification System	Classification Symbols	
Int.C1.5	C 11 D	
Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched ⁸		
III. DOCUMENTS CONSIDERED TO BE RELEVANT⁹		
Category ^o	Citation of Document, ¹¹ with indication, where appropriate, of the relevant passages ¹²	Relevant to Claim No. ¹³
X	EP-A-0 346 994 (UNILEVER) 20 December 1989, see the whole document ---	1-10
X	EP-A-0 359 308 (UNILEVER) 21 March 1990, see page 3, line 35 - page 8, line 56; claims ---	1-3, 8-10
X	EP-A-0 346 993 (UNILEVER) 20 December 1989, see the whole document ---	1-10
X	FR-A-2 390 497 (COLGATE-PALMOLIVE) 8 December 1978, see page 2, lines 31-40; examples; claims, & US-A-4 2448 840 (cited in the application) ---	1,2
Y	EP-A-0 120 638 (ALBRIGHT & WILSON) 3 October 1984, see the whole document --- -/-	1-3
<div style="display: flex; justify-content: space-between;"> <div style="width: 45%;"> <p>^o Special categories of cited documents : ¹⁰</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> </div> <div style="width: 45%;"> <p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step</p> <p>"Y" document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.</p> <p>"&" document member of the same patent family</p> </div> </div>		
IV. CERTIFICATION		
Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report	
09-08-1991	11 SEP 1991	
International Searching Authority	Signature of Authorized Officer	
EUROPEAN PATENT OFFICE	 P. TAZELANS	

III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)		
Category °	Citation of Document, with indication, where appropriate, of the relevant passages	Relevant to Claim No.
Y	FR-A-2 325 639 (ALBRIGHT & WILSON) 22 April 1977, see the whole document ---	1-3
Y	GB-A-2 021 141 (ALBRIGHT & WILSON) 28 November 1979, see the whole document -----	1-3

**ANNEX TO THE INTERNATIONAL SEARCH REPORT
ON INTERNATIONAL PATENT APPLICATION NO.**

EP 9100768

SA 46813

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report. The members are as contained in the European Patent Office EDP file on 06/09/91. The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP-A- 0346994	20-12-89	AU-A- 3626789	14-12-89
		AU-A- 3626989	14-12-89
		AU-A- 3632989	14-12-89
		EP-A- 0346993	20-12-89
		EP-A- 0346995	20-12-89
		JP-A- 2034699	05-02-90
		JP-A- 2034700	05-02-90
		JP-A- 2038500	07-02-90
EP-A- 0359308	21-03-90	AU-A- 4146589	22-03-90
		JP-A- 2133498	22-05-90
EP-A- 0346993	20-12-89	AU-A- 3626789	14-12-89
		AU-A- 3626989	14-12-89
		AU-A- 3632989	14-12-89
		EP-A- 0346994	20-12-89
		EP-A- 0346995	20-12-89
		JP-A- 2034699	05-02-90
		JP-A- 2034700	05-02-90
		JP-A- 2038500	07-02-90
FR-A- 2390497	08-12-78	GB-A- 1565735	23-04-80
		AT-B- 375956	25-09-84
		AU-B- 522490	10-06-82
		AU-A- 3591378	15-11-79
		BE-A- 866894	01-09-78
		CA-A- 1097554	17-03-81
		CH-A- 640881	31-01-84
		DE-A, C 2819975	16-11-78
		NL-A- 7805052	14-11-78
		SE-B- 437673	11-03-85
		SE-A- 7805236	11-11-78
		US-A- 4244840	13-01-81
EP-A- 0120638	03-10-84	AU-B- 559302	05-03-87
		AU-A- 2549984	13-09-84
		GB-A, B 2136444	19-09-84
FR-A- 2325639	22-04-77	GB-A- 1488352	12-10-77

**ANNEX TO THE INTERNATIONAL SEARCH REPORT
ON INTERNATIONAL PATENT APPLICATION NO.**

EP 9100768

SA 46813

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report. The members are as contained in the European Patent Office EDP file on 06/09/91
The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
FR-A- 2325639		AU-A- 7736575	22-07-76
		DE-A, C 2501622	24-07-75
		US-A- 4191704	04-03-80

GB-A- 2021141	28-11-79	None	

THIS PAGE BLANK (USPTO)